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(54) **DRY, WATER-SOLUBLE POWDER, SUBSTITUTED HETEROCYCLIC ACID OR SUBSTITUTED PHENOL HERBICIDAL COMPOSITIONS, AND METHOD OF PREPARING SAME**

HERBIZIDE ZUSAMMENSETZUNGEN IN FORM TROCKENER, WASSERLÖSLICHER PULVER AUF DER BASIS SUBSTITUIERTER PHENOLE ODER HETEROZYKLISCHER SÄUREN UND VERFAHREN ZU IHRER HERSTELLUNG

COMPOSITIONS HERBICIDES EN POUDRE SECHE SOLUBLE DANS L'EAU, A L'ACIDE HETEROCYCLIQUE SUBSTITUE OU AU PHENOL SUBSTITUE, ET LEUR PROCEDE DE PREPARATION

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(56) References cited:
EP-A- 0 368 806
US-A- 4 015 970
US-A- 5 022 917
WO-A-93/03614
US-A- 4 332 614
US-A- 5 160 528

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Description

Related Applications:

[0001] This application is based on a continuation-in-part of Application Serial No. 07/745,866, filed August 16, 1991, now U.S. Letters Patent No. 5,221,319; Application Serial No. 07/911,757, filed July 10, 1992, which is now abandoned; and pending Application Serial No. 07/928,132, filed August 10, 1992, which has been published as WO 93/03614.

Background of the Invention

1. Field of the Invention

[0002] This invention relates to water-soluble herbicidal compositions in dry powdered form which include herbicidally active agents selected from the group consisting of 3,6-dichloropicolinic acid, 3,5,6-trichloro-2-pyridyloxyacetic acid, 4-amino-3,5,6-trichloropicolinic acid, 3,7-dichloro-8-quinolinecarboxylic acid, 3-isopropyl-1H-2,1,3-benzothiadiazin-4 (3H)-one 2,2-dioxide, 3,5-dibromo-4-hydroxybenzonitrile, 3,5-diiodo-4-hydroxybenzonitrile, each of which alone, or in combination with one another, are not readily soluble in water.

2. Description of the Prior Art

[0003] Those herbicidal agents registered for use by commercial applicators are generally sold in concentrated form for economy of transport, and then diluted by the applicator either at a central distribution center, or less frequently at the point of use. The majority of effective herbicides and plant growth regulators are foliarly absorbed and therefore, to be effective, must be applied to the foliage of the target pest species. Other herbicides are root absorbed and the product must be applied in a manner as to be available to the roots of the target. This is commonly accomplished by spraying a dilute water solution, or dispersion of the desired pesticide on the vegetation to be treated. Most herbicides are therefore marketed as either (1) liquid or dry water-soluble formulations, (2) liquid, water emulsifiable formulations, or (3) solid or liquid water dispersible formulations. The concentrated formulations are diluted to the required effective concentration by the person doing the spray application. Thus, in order to obtain optimum effectiveness and to minimize agitation and other mechanical suspension requirements, water-soluble formulations are normally preferred.

[0004] Because of the difficulties of manufacturing a dry, soluble form of herbicide, most dry formulations are simply dispersible forms of essentially insoluble active ingredients. Typical examples of formulations are (1) wettable powders, (2) water dispersible granules, or (3) dry flowables. Formulations of these types depend heavily on surfactants and grinding techniques to provide a dry formulation of active ingredients that can be temporarily dispersed or suspended in water for spray application. Even when a dispersion can initially be obtained in water, the time of full dispersion is usually limited, thus requiring stirring, agitation with air, or other mechanical mixing. Dispersions of this type present additional problems in that the material tends to clog spray nozzles and other distribution components, and require the user to prepare smaller than desired batches in order to minimize application problems.

[0005] Because of the problems associated with attempting to prepare the dry powdered herbicide, suppliers have resorted in some instances to dissolution of the active ingredient in an organic solvent such as mineral spirits or the like. The concentrated formulation, containing suitable surfactants, is then diluted with water to form a dispersion that again usually necessitates some type of agitation to maintain the phases substantially homogeneous for a useful period of time.

[0006] In instances where the herbicides are dissolved in a solvent for shipment as a concentrate, the solvent presents health and physical hazards to the manufacturer as well as the user, the solvents add to the overall cost of the product, and the solvent agent is oftentimes phytotoxic to desirable plant species.

[0007] The wettable powders and solvent dissolved herbicides are frequently packaged in plastic containers and disposal of these plastic packages is becoming increasingly difficult from an environmental standpoint.

[0008] Substituted phenoxy and/or benzoic acid herbicides such as (2,4-dichlorophenoxy)acetic acid [2,4-D], 4-(2,4-dichlorophenoxy)butanoic acid [2,4-DB], (±)-2-(4-chloro-2-methylphenoxy)propanoic acid [MCPP], (4-chloro-2-methylphenoxy)acetic acid [MCPA], (±)-2-(2,4-dichlorophenoxy)propanoic acid [dichlorprop], 3,6-dichloro-2-methoxybenzoic acid [dicamba], 3-amino-2,5-dichlorobenzoic acid [chloramben], and 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoic acid [acifluorfen] have long been used to control unwanted vegetation.

[0009] These substituted phenoxy and/or benzoic acid herbicides are white crystalline solids with very low vapor pressures and low water solubilities. They are soluble only in alkaline solutions or polar organic solvents.

[0010] Phenoxy and/or benzoic acid herbicides are available commercially as acid, ester, alkali metal, and amine salt formulations that can also be applied as mixtures with other herbicides. The alkali metal and especially the amine salt formulations are preferred because they are the most water-soluble and can be more readily applied as aqueous

sprays. However, the esters must be applied either as emulsions in water, or as solutions in organic solvents such as oils. 2,4-D for example, is an insoluble crystalline material having a pK_a of approximately 2.6. For ease of application, 2,4-D is normally converted to a water-soluble amine or mineral salt by the manufacturer and then dissolved by the applicator in a water carrier before use.

[0011] However, water-soluble substituted phenoxy and/or benzoic acid salts exhibiting herbicidal activity are difficult to prepare in a dry state. Soluble salts such as potassium or sodium or dimethylamine must be first prepared in water or a solvent and then the solvent removed. This requires special equipment, is energy intensive, and frequently generates undesirable waste products. As a consequence, most dry forms of herbicide that are marketed are not of a soluble type but rather are merely dispersible forms of the essentially insoluble herbicide acid which are distributed as a wettable powder or a wettable, dispersible granule. Although both inorganic and organic salt forms are commercially available, the most common salt form is the dimethylamine salt of the substituted phenoxy or substituted benzoic acid herbicide. Typical formulations range from about 20% to 50% active ingredient concentrations in water or solvent solutions. WO 93/03614 discloses dry, water-soluble phenoxy and benzoic acid herbicides and method of their preparation.

[0012] Similarly, 3,6-dichloropicolinic acid as a monoethanolamine salt [clopyralid], 3,5,6-trichloro-2-pyridyloxyacetic acid as a triethylamine salt [triclopyr], 4-amino-3,5,6-trichloropicolinic acid as a potassium salt [picloram], 3,7-dichloro-8-quinolinecarboxylic acid as a 50% wettable powder [quinclorac] and 3-isopropyl-1H-2,1,3-benzothiadiazin-4 (3H)-one 2,2-dioxide as a sodium salt [bentazon] have been used commercially as herbicidal compositions.

[0013] Picloram has been combined with the amine salt of 2,4-D acid, clopyralid has been combined with the amine salt of 2,4-D acid, and triclopyr plus 2,4-D acid as an EC ester, have all been suggested and are in use as herbicidal agents.

[0014] Wettable powders, water-soluble amine salts, oil soluble amine salts, and emulsifiable concentrates have also been provided of substituted phenolic herbicides including a 33.4% octanoic acid ester of 3,5-dibromo-4-hydroxybenzonitrile [bromoxynil], a 31.7% octanoic acid ester of bromoxynil plus 34% of isooctyl ester of 2-methyl-chlorophenoxyacetic acid, bromoxynil plus MCPA, and the octanoic acid ester of 3,5-diiodo-4-hydroxybenzonitrile [ioxynil]. Various combinations of ioxynil and/or bromoxynil have been marketed in various foreign countries.

[0015] The water solubility of a solid herbicidal phenol such as bromoxynil is no more than about 130 mg/L. The water solubility of ioxynil is no greater than about 50 mg/L.

[0016] EP-A-368 806 discloses preparation of dry powders of the phenolic herbicides bromoxynil and ioxynilusing salts of weak acids.

[0017] All of these herbicidal compositions suffer from the disadvantage that the acids either have to be converted to water-soluble form by forming amine salts therefrom, or other equivalent treatment, before they may be used by an applicator.

Summary of the Invention

[0018] The present invention relates to a method of preparing dry, water-soluble herbicide powder compositions consisting essentially of a solid herbicidal agent selected from the group consisting of 3,6-dichloropicolinic acid, 3,5,6-trichloro-2-pyridyloxyacetic acid, 4-amino-3,5,6-trichloropicolinic acid, 3,7-dichloro-8-quinolinecarboxylic acid, 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide, 3,5-dibromo-4-hydroxybenzonitrile, 3,5-diiodo-4-hydroxy-benzonitrile, and a solubilization medium. The invention also concerns said herbicide powder compositions, which may be dissolved in water at concentrations providing from at least about 0.1% to at least least about 2-1/2% by weight of the active herbicide in the final herbicidal solution.

[0019] A dry herbicidal product is preferred over a liquid concentrated herbicide for a number of reasons. Dry formulations are more stable to temperature variations encountered in storage and shipping. For example, freezing can destabilize liquid products interfering with the effectiveness of the herbicide. Similarly, high storage temperatures can lead to solvent losses when an organic solvent is used to dissolve the agent or cause hydrolysis when water is a solvent, thereby adversely affecting active ingredient concentrations.

[0020] Dry herbicidal formulations are less dangerous than liquid products. Package leakage during handling is much less likely. If accidentally punctured, dry package leakage is much less severe and easier to clean up than a liquid product. Also, personnel protection is easier to accomplish with dry products because the material cannot as easily splash into the eyes or skin of the applicator. Spills of solvent containing formulations are potentially flammable, further militating against the use of solvents for dissolving the herbicide.

[0021] The ease of packaging is significantly enhanced with dry products over liquid formulations and packaging flexibilities are greatly enhanced. Paper containers or wax treated packages can be used, as well as plastic containers. With liquids, specially treated plastic containers or glass containers are normally required. Paper or cardboard packages can be compressed and disposed of much easier than plastic, glass or metal containers.

[0022] Transportation costs of dry products are potentially less expensive than is the case where a liquid carrier must

also be transported. Although a number of concentrated liquid products having a fairly high active ingredient content are in commercial use, many formulations are sold in the 20-30% active ingredient range with the remainder of the product being water along with a small amount of dispersing agents or product appearance or handling enhancers.

[0023] The dry, water-soluble herbicide powder compositions of the subject invention are prepared by dry blending the herbicidal agent with a quantity of solid, substantially anhydrous diammonium phosphate (DAP), dipotassium phosphate (DPP), disodium phosphate (DSP), trisodium phosphate (TSP), tripotassium phosphate (TPP), and mixtures thereof which serves as a solubilization medium for the herbicidal agent. A sufficient amount of the DAP, DPP, DSP, TSP or TPP solubilization medium is provided in the dry blended mixture in relationship to the quantity of herbicidal agent combined therewith to cause the dry blended mixture to substantially dissolve in water during preparation of a herbicidal solution which contains an adequate proportion of the herbicidal agent to provide from about 0.1% to about 2-1/2% by weight of the active herbicide in the herbicidal solution.

[0024] A sufficient amount of the DAP, DPP, DSP, TSP or TPP solubilization medium is dry blended with the dry substituted heterocyclic acid or phenolic herbicidal agent, or a heterocyclic acid and phenolic herbicide with a phenoxy or benzoic acid herbicide to provide at least about 1 mole and to about 5 moles of the phosphate medium for each mole of active herbicidal agent in the dry blended mixture.

[0025] The dry, water-soluble herbicides of the subject invention are easily manufactured by dry blending the ingredients in powdered form. No unusual manufacturing techniques are required such as grinding to very fine sizes or classification procedures normally necessary to obtain a suitable dispersible product. Product raw material costs are comparable to widely used organic amine or salts of substituted heterocyclic or substituted phenol herbicide formulations currently being marketed. The raw materials are all commercially available and readily obtainable at competitive prices.

Detailed Description of the Invention

[0026] A quantity of a substantially solid herbicidal agent selected from the group consisting of 3,6-dichloropicolinic acid, 3,5,6-trichloro-2-pyridyloxyacetic acid, 4-amino-3,5,6-trichloropicolinic acid, 3,7-dichloro-8-quinolinecarboxylic acid, 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide, 3,5-dibromo-4-hydroxybenzonitrile, 3,5-diiodo-4-hydroxy-benzonitrile, each of which alone, or in combination with one another, are not readily soluble in water is added to an amount of a substantially solid solubilization medium for the herbicidal agent. The preferred solubilization agent is a quantity of solid, substantially anhydrous diammonium phosphate (DAP), dipotassium phosphate (DPP), disodium phosphate (DSP), trisodium phosphate (TSP), tripotassium phosphate (TPP), and mixtures thereof which serves as a solubilization medium for the herbicidal agent.

[0027] Preferred formulations include at least about 25 weight percent of the active herbicidal acid, and in certain instances about 50 weight percent of the active herbicidal agent or combinations thereof.

[0028] The herbicidal agent and the phosphate solubilization medium are dry blended to produce a substantially homogeneous mixture thereof. This dry blended mixture may be packaged in paper containers, or other suitable packages, without further processing such as pulverization, extended grinding, or critical classification. However, the particle size is preferably small enough to permit relatively rapid wetting when added to water. At least about 1 mole to about 5 moles of the solubilization medium is provided for each mole of active herbicidal agent in the dry blended mixture.

EXAMPLE I

[0029] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Clopyralid acid Tech. Grade - (95%)	50.00	52.63	7.89	49.92
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		2.00	0.30	
5. DAP - Diammonium Phosphate-Reagent Grade - 98%		43.37	6.51	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-5 of Example I were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example I was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE II

[0030] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Triclopyr acid Tech. Grade - (98%)	50.00	51.02	7.65	50.11
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
5. DAP - Diammonium Phosphate-Reagent Grade - 98%		41.98	6.30	
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example I. Water solubility and oven stability were, likewise, similar.

EXAMPLE III

[0031] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 10 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Bentazon acid Tech. Grade - (92.4%)	50.00	54.11	5.41	52.42
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.10	
3. Sorbitan tristearate (SPAN-65)		1.00	0.10	
4. Spray dry synthetic silica (Wessalon 50-S)		3.00	0.30	
5. DAP - Diammonium Phosphate - Reagent Grade - 98%		40.89	4.09	
TOTALS		100.00%	10.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example I. Water solubility and oven stability were, likewise, similar.

EXAMPLE IV

[0032] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Picloram acid Tech. Grade - (95%)	50.00	52.63	7.89	49.18
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
5. TSP - Trisodium Phosphate - (Anhyd)		40.37	6.06	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example I. Water solubility and oven stability were, likewise, similar.

EXAMPLE V

[0033] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Quinclorac acid Tech. Grade - (94%)	25.00	26.60	3.99	24.85
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
5. TSP - Trisodium Phosphate - (Anhyd)		66.40	9.96	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-5 of Example V were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 25% active herbicide powder prepared in accordance with Example V was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE VI

[0034] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
Bentazon acid Tech. Grade - (94%)	33.33	35.46	5.32	33.96

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Acifluorfen acid Tech.Grade - (92%)	16.67	18.12	2.72	16.66
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
5. DAP - Diammonium Phosphate - Reagent Grade - 98%		39.42	5.91	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-6 of Example VI were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example VI was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE VII

[0035] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Triclopyr acid Tech. Grade - (98%)	16.67	17.01	2.55	17.25
2. 2,4-D acid Tech. Grade - (97%)	33.33	34.36	5.15	33.87
3. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
4. Sorbitan tristearate (SPAN-65)		1.00	0.15	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
5. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
6. DAP - Diammonium Phosphate - Reagent Grade - 98%		41.63	6.25	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-6 of Example VII were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example VII was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE VIII

[0036] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Picloram acid Tech. Grade - (95%)	10.00	10.53	1.58	10.08
2. 2,4-D acid Tech. Grade - (97%)	40.00	41.24	6.19	40.27
3. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
4. Sorbitan tristearate (SPAN-65)		1.00	0.15	
5. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
6. TSP - Trisodium Phosphate - (Anhyd)		41.23	6.18	
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example VII. Water solubility and oven stability were, likewise, similar.

EXAMPLE IX

[0037] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Clopyralid acid Tech. Grade - (95%)	8.00	8.42	1.26	8.09
2. 2,4-D acid Tech. Grade - (97%)	42.00	43.30	6.50	41.81
3. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
4. Sorbitan tristearate (SPAN-65)		1.00	0.15	
5. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
6. DSP - Disodium Phosphate - (Anhyd)		41.28	6.19	
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example VII. Water solubility and oven stability were, likewise, similar.

EXAMPLE X

[0038] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Quinclorac acid Tech. Grade - (94%)	13.33	14.18	2.13	14.35
2. Dichlorprop acid Tech.Grade - (94%)	26.67	28.37	4.26	27.28
3. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
4. Sorbitan tristearate (SPAN-65)		1.00	0.15	
5. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
6. TSP - Trisodium Phosphate - (Anhyd)		50.45	7.56	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-6 of Example X were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example X was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XI

[0039] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. 2,4-D acid Tech. Grade - (97%)	31.25	32.22	4.83	30.66
2. MCPP acid Tech. Grade - (Dry, 95%)	15.62	16.44	2.47	15.76
3. Clopyralid Acid - Tech. Grade - (95%)	3.13	3.29	0.49	3.36
4. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
5. Sorbitan tristearate (SPAN-65)		1.00	0.15	
6. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
7. DAP - Diammonium Phosphate-Reagent Grade - 98%		41.05	6.16	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-7 of Example XI were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example XI was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XII

[0040] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. 2,4-D acid Tech. Grade - (97%)	31.25	32.22	4.83	31.48
2. MCPP acid Tech. Grade - (Dry, 95%)	15.62	16.44	2.47	15.83
3. Triclopyr Acid - Tech. Grade - (98%)	3.13	3.19	0.48	3.13
4. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
5. Sorbitan tristearate (SPAN-65)		1.00	0.15	
6. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
7. DAP - Diammonium Phosphate - Reagent Grade - 98%		41.15	6.17	
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example XI. Water solubility and oven stability were, likewise, similar.

EXAMPLE XIII

[0041] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. 2,4-D acid Tech. Grade - (97%)	31.25	32.22	4.83	32.07
2. MCPP acid Tech. Grade - (Dry, 95%)	15.16	16.44	2.47	15.72
3. Picloram Acid - Tech. Grade - (95%)	3.13	3.29	0.49	3.25
4. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
5. Sorbitan tristearate (SPAN-65)		1.00	0.15	
6. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
7. DAP - Diammonium Phosphate - Reagent Grade - 98%		41.05	6.16	
TOTALS		100.00%	15.00 gms.	

The 50% active herbicide powder was prepared in accordance with Example XI. Water solubility and oven stability were, likewise, similar.

EXAMPLE XIV

[0042] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. MCPA acid Tech. Grade - (95%)	33.34	35.09	5.26	33.47
2. MCPP acid Tech. Grade - (Dry, 95%)	13.33	14.03	2.10	13.13
3. Triclopyr Acid - Tech. Grade - (98%)	3.33	3.40	0.51	3.00
4. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
5. Sorbitan tristearate (SPAN-65)		1.00	0.15	
6. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
7. TPP-Tripotassium Phosphate - (Anhyd)		40.48	6.08	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-7 of Example XIV were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example XIV was added to a glass graduated cylinder which contained

98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XV

[0043] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. 2,4-D acid Tech. Grade - (97%)	22.23	22.92	3.44	22.37
2. Dichlorprop acid Tech.Grade - (95%)	22.22	23.39	3.51	23.53
3. Triclopyr Acid - Tech. Grade - (98%)	5.55	5.66	0.85	4.93
4. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
5. Sorbitan tristearate (SPAN-65)		1.00	0.15	
6. Spray dry synthetic silica (Wessalon 50-S)		5.00	0.75	
7. DPP - Dipotassium Phosphate - (Anhyd)		41.03	6.15	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-7 of Example XV were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 50% active herbicide powder prepared in accordance with Example XV was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XVI

[0044] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 20.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Bromoxynil Tech. Grade-96.9%	35.00	36.12	7.22	35.77

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 20.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.20	
3. Sorbitan tristearate (SPAN-65)		1.00	0.20	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	1.00	
5. TPP-Tripotassium phosphate (Anhyd)		56.88	11.38	
TOTALS		100.00%	20.00 gms.	

Ingredients 1-5 of Example XVI were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 35% active herbicide powder prepared in accordance with Example XVI was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XVII

[0045] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 20.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Bromoxynil Tech. Grade-96.9%	30.00	30.96	6.19	30.75
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.20	
3. Sorbitan tristearate (SPAN-65)		1.00	0.20	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	1.00	
5. DSP-Disodium Phosphate (Anhyd)		62.04	12.41	
TOTALS		100.00%	20.00 gms.	

The 30% active herbicide powder was prepared in accordance with Example XVI. Water solubility and oven stability

were, likewise, similar.

EXAMPLE XVIII

[0046] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 20.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Bromoxynil Tech. Grade-96.9%	25.00	25.80	5.16	25.00
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.20	
3. Sorbitan tristearate (SPAN-65)		1.00	0.20	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	1.00	
5. TSP-Trisodium phosphate (Anhyd)		67.20	13.44	
TOTALS		100.00%	20.00 gms.	

The 25% active herbicide powder was prepared in accordance with Example XVI. Water solubility and oven stability were, likewise, similar.

EXAMPLE XIX

[0047] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Ioxynil Tech. Grade-97%	40.00	41.24	6.19	39.30
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		2.00	0.30	
5. TSP-Trisodium phosphate (Anhyd)		54.76	8.21	
TOTALS		100.00%	15.00 gms.	

Ingredients 1-5 of Example XIX were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 40% active herbicide powder prepared in accordance with Example XIX was added to a glass graduated cylinder which contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

EXAMPLE XX

[0048] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Ioxynil Tech. Grade-97%	35.00	36.08	5.41	34.00
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	
4. Spray dry synthetic silica (Wessalon 50-S)		2.00	0.30	
5. DPP-Dipotassium phosphate (Anhyd)		59.92	8.99	
TOTALS		100.00%	15.00 gms.	

The 35% active herbicide powder was prepared in accordance with Example XIX. Water solubility and oven stability were, likewise, similar.

EXAMPLE XXI

[0049] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. Ioxynil Tech. Grade-97%	25.00	25.77	3.87	25.00
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.15	
3. Sorbitan tristearate (SPAN-65)		1.00	0.15	

(continued)

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 15.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
4. Spray dry synthetic silica (Wessalon 50-S)		2.00	0.30	
5. DAP-Diammonium phosphate-Reagent Grade - 98%		70.23	10.53	
TOTALS		100.00%	15.00 gms.	

[0050] The 25% active herbicide powder was prepared in accordance with Example XIX. Water solubility and oven stability were, likewise, similar.

[0051] The prepared aqueous sample containing 2.0 weight percent of the 25% to 50% active herbicide powder in all instances yielded a substantially clear solution within two minutes. It was not necessary to heat or agitate the product to maintain the ingredients in solution while sitting on a shelf for several days at room temperature. Tests of the dry blended material dissolved in water yielded clear solutions at levels of 0.5%, 1%, 2%, and 4 weight percent. Weight percent in this respect means 1 gram of the active dry powder for each 99 milliliters of tap water. Normal herbicide application concentrations range from about 1/2 weight percent of the active acid to about 2% of the active acid.

EXAMPLE XXII

[0052] The following ingredients were dry blended:

Ingredients	% Active Ingredients	Weight % Reagents	Makeup for Grinding of 20.0 Grams - In Grams	HPLC (High Performance Liquid Chromatography Analysis)
1. MCPA acid Tech. Grade-(94%)	20.00	21.28	4.26	20.75
2. Bromoxynil Tech. Grade-(96.9%)	20.00	20.64	4.13	20.33
2. Sodium napthalenesulfonate formaldehyde polymer (Lomar PW)		1.00	0.20	
3. Sorbitan tristearate (SPAN-65)		1.00	0.20	
4. Spray dry synthetic silica (Wessalon 50-S)		5.00	1.00	
5. TSP-Trisodium phosphate-(Anhyd)		51.08	10.21	
TOTALS		100.00%	20.00 gms.	

[0053] Ingredients 1-6 of Example XXII were ground to a fine, dry powder (averaging about 50 microns particle size) in a laboratory analytical mill. The mixture was transferred to a capped glass container. A 2 gm. sample of the 40% active herbicide powder prepared in accordance with Example XXII was added to a glass graduated cylinder which

contained 98 ml. of city water. The stoppered cylinder was inverted several times over a period of 5 minutes. The 2 weight percent of herbicidal agent added to the water dissolved, with only a small quantity of silica remaining as a dispersion. After 96 hours in an oven at 50°C, a sample of the powder indicated no visible changes.

[0054] It has been determined that the best results are obtained when an anhydrous DAP, DPP and DSP are utilized as the solubilization medium for the substituted phenoxy and/or benzoic acid herbicidal agent, with the preferred medium being DAP. In all cases, the anhydrous DAP, DPP, DSP, TSP or TPP solubilization medium should contain less than about 1% water by weight.

Claims

1. A dry, water-soluble herbicide powder composition consisting essentially of:
a dry blended admixture of

a first quantity of dry, powder particles of a substantially solid herbicidal agent selected from the group consisting of 3,6-dichloropicolinic acid, 3,5,6-trichloro-2-pyridyloxyacetic acid, 4-amino-3,5,6-trichloropicolinic acid, 3,7-dichloro-8-quinolinecarboxylic acid, 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide, 3,5-dibromo-4-hydroxybenzonitrile, 3,5-diiodo-4-hydroxybenzonitrile, each of which alone, or in combination with one another, are not readily soluble in water, and

a second quantity of dry, solid, powder particles selected from the group consisting of diammonium phosphate, dipotassium phosphate, disodium phosphate, trisodium phosphate, tripotassium phosphate, and mixtures thereof as a solubilization medium for the herbicidal agent,

said first and second quantities of the herbicidal agent and the solubilization medium being the predominate constituents of the composition,

there being at least about 1 mole of solubilization medium for each mole of herbicidal agent,

said herbicidal agent and the solubilization medium having been dry blended in powdered form without changing the physical state of the particles to retain the discrete particulate character of each of said first and second quantities of said particles, and in the absence of chemical reaction between said herbicidal agent and said solubilization medium to form a relatively uniform dry mixture thereof,

there being a sufficient quantity of the phosphate medium in the dry blended mixture in relationship to the quantity of herbicidal agent combined therewith such that the dry blended powder mixture will dissolve in water during preparation of a herbicidal solution therefrom that contains an adequate proportion of the herbicidal agent to provide from about 0.1 to about 2-1/2% by weight of the active herbicidal agent in the herbicidal solution.

2. A herbicide as set forth in Claim 1 wherein said dry solubilization medium is diammonium phosphate.

3. A herbicide as set forth in Claim 1 wherein said dry solubilization medium is dipotassium phosphate.

4. A herbicide as set forth in Claim 1 wherein said dry solubilization medium is trisodium phosphate.

5. A herbicide as set forth in Claim 1 wherein said dry, solid, powder herbicide is 3,5-diiodo-4-hydroxy-benzonitrile and 3,5-dibromo-4-hydroxybenzonitrile.

6. A method of preparing a dry, water-soluble, herbicide powder composition consisting essentially of the steps of:

providing a first quantity of dry, powder particles of a substantially solid herbicidal agent selected from the group consisting of 3,6-dichloropicolinic acid, 3,5,6-trichloro-2-pyridyloxyacetic acid, 4-amino-3,5,6-trichloropicolinic acid, 3,7-dichloro-8-quinolinecarboxylic acid, 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide, 3,5-dibromo-4-hydroxybenzonitrile, 3,5-diiodo-4-hydroxybenzonitrile, each of which alone, or in combination with one another, are not readily soluble in water,

providing a second quantity of dry, solid powder particles of diammonium phosphate as a solubilization medium

for the herbicidal agent,

said first and second quantities of the herbicidal agent and the solubilization medium being the predominate constituents of the composition,

there being at least 1 mole of solubilization medium for each mole of herbicidal agent; and

dry blending the herbicidal agent and the solubilization medium in powdered form without changing the physical state of the particles to retain the discrete particulate character of each of said first and second quantities of said particles, and in the absence of chemical reaction between said herbicidal agent and said solubilization medium to produce a relatively uniform dry mixture thereof,

a sufficient quantity of the phosphate solubilization medium being provided in the dry blended mixture in relationship to the quantity of herbicidal agent combined therewith to cause the powdered dry blended mixture to substantially dissolve in water during preparation by the applicator of a herbicidal solution which contains an adequate proportion of the herbicidal agent to provide from about 0.1% to about 2-1/2% by weight of the active herbicidal agent in the herbicidal solution.

7. A method as set forth in Claim 6 wherein is included the step of providing a mixture of said herbicidally active agents for dry blending with the phosphate medium.

8. A method as set forth in Claim 6 wherein is included the step of incorporating an anti-caking agent in the dry blended mixture.

9. A method of preparing a herbicide solution consisting essentially of the steps of:

adding to a volume of water, a dry powdered herbicidal composition according to claim 1;

adding a sufficient quantity of the powdered dry blended admixture of the herbicidal agent and the phosphate solubilization medium to the volume of water to cause the powdered dry blended mixture dissolved in the water to provide from about 0.1% to about 2-1/2% by weight of the active herbicidal agent in the herbicidal solution; and

agitating the water containing the herbicidal agent and the solubilization medium for a time period sufficient to effect substantial dissolution of the agent and the medium in the volume of water.

Patentansprüche

1. Trockene wasserlösliche Herbizid-Pulver-Zusammensetzung, im wesentlichen bestehend aus:

einer trocken gemischten Mischung von

einer ersten Menge von trockenen Pulverteilchen eines im wesentlichen festen herbiziden Mittels, ausgewählt aus der Gruppe bestehend aus 3,6-Dichlorpicolinsäure, 3,5,6-Trichlor-2-pyridyloxyessigsäure, 4-Amino-3,5,6-trichlorpicolinsäure, 3,7-Dichlor-8-chinolincarbonsäure, 3-Isopropyl1H-2,1,3-benzothiadiazin-4(3H)-on-2,2-dioxid, 3,5-Dibrom-4-hydroxybenzonitril, 3,5-Diiod-4-hydroxybenzonitril, von denen jedes allein oder in Kombination mit einander in Wasser nicht leicht löslich ist, und

einer zweiten Menge von trockenen festen Pulverteilchen, ausgewählt aus der Gruppe bestehend aus Diammoniumphosphat, Dikaliumphosphat, Dinatriumphosphat, Trinatriumphosphat, Trikaliumphosphat und Mischungen davon, als Solubilisierungsmedium für das herbizide Mittel,

wobei die ersten und zweiten Mengen des herbiziden Mittels und des Solubilisierungsmediums die überwiegenden Bestandteile der Zusammensetzung sind,
wobei mindestens 1 mol Solubilisierungsmedium für jedes mol herbizides Mittel vorhanden ist,
wobei das herbizide Mittel und das Solubilisierungsmedium in pulverisierter Form trocken gemischt worden sind, ohne den körperlichen Zustand oder Aggregatzustand der Teilchen zu verändern, um den diskreten teilchenför-

migen Charakter von jeder der ersten und zweiten Mengen der Teilchen zu bewahren, und in Abwesenheit von einer chemischen Reaktion zwischen dem herbiziden Mittel und dem Solubilisierungsmedium, um eine relativ gleichförmige trockene Mischung davon zu bilden,
wobei eine ausreichende Menge des Phosphatmediums in der trocken gemischten Mischung in Bezug auf die Menge des damit kombinierten herbiziden Mittels vorhanden ist, so dass die trocken gemischte Pulvermischung sich in Wasser lösen wird während der Herstellung einer herbiziden Lösung daraus, die einen adäquaten Anteil des herbiziden Mittels enthält, um ungefähr 0,1 bis ungefähr 2-1/2 Gew.-% des herbiziden Wirkstoffs in der herbiziden Lösung bereitzustellen.

2. Herbizid nach Anspruch 1, wobei das trockene Solubilisierungsmedium Diammoniumphosphat ist.

3. Herbizid nach Anspruch 1, wobei das trockene Solubilisierungsmedium Dikaliumphosphat ist.

4. Herbizid nach Anspruch 1, wobei das trockene Solubilisierungsmedium Trinatriumphosphat ist.

5. Herbizid nach Anspruch 1, wobei das trockene feste Pulver-Herbizid 3,5-Diiod-4-hydroxybenzonitril und 3,5-Dibrom-4-hydroxybenzonitril ist.

6. Verfahren zum Herstellen einer trockenen, wasserlöslichen Herbizid-Pulver-Zusammensetzung, bestehend im wesentlichen aus den Schritten:

Bereitstellen einer ersten Menge von trockenen Pulverteilchen eines im wesentlichen festen herbiziden Mittels, ausgewählt aus der Gruppe bestehend aus 3,6-Dichlorpicolinsäure, 3,5,6-Trichlor-2-pyridyloxyessigsäure, 4-Amino-3,5,6-trichlorpicolinsäure, 3,7-Dichlor-8-chinolincarbonsäure, 3-Isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-on-2,2-dioxid, 3,5-Dibrom-4-hydroxybenzonitril, 3,5-Diiod-4-hydroxybenzonitril, von denen jedes allein oder in Kombination mit einander in Wasser nicht leicht löslich ist, und

Bereitstellen einer zweiten Menge von trockenen festen Pulverteilchen von Diammoniumphosphat als Solubilisierungsmedium für das herbizide Mittel,

wobei die ersten und zweiten Mengen des herbiziden Mittels und des Solubilisierungsmediums die überwiegenden Bestandteile der Zusammensetzung sind,
wobei mindestens 1 mol Solubilisierungsmedium für jedes mol herbizides Mittel vorhanden ist; und

trockenes Mischen des herbiziden Mittels und des Solubilisierungsmediums in pulverisierter Form, ohne den körperlichen Zustand oder Aggregatzustand der Teilchen zu verändern, um den diskreten teilchenförmigen Charakter von jeder der ersten und zweiten Mengen der Teilchen zu bewahren, und in Abwesenheit von einer chemischen Reaktion zwischen dem herbiziden Mittel und dem Solubilisierungsmedium, um eine relativ gleichförmige trockene Mischung davon zu bilden,

wobei eine ausreichende Menge des Phosphat-Solubilisierungsmediums in der trocken gemischten Mischung in Bezug auf die Menge des damit kombinierten herbiziden Mittels bereitgestellt wird, so dass bewirkt wird, dass die pulverförmige, trocken gemischte Mischung sich in Wasser im wesentlichen löst während der durch die Applikatorvorrichtung erfolgenden Herstellung einer herbiziden Lösung, die einen adäquaten Anteil des herbiziden Mittels enthält, um ungefähr 0,1 Gew.-% bis ungefähr 2-1/2 Gew.-% des herbiziden Wirkstoffs in der herbiziden Lösung bereitzustellen.

7. Verfahren nach Anspruch 6, worin der Schritt enthalten ist, eine Mischung der herbiziden Wirkstoffe zum trockenen Mischen mit dem Phosphatmedium bereitzustellen.

8. Verfahren nach Anspruch 6, worin der Schritt enthalten ist, ein Mittel zum Verhindern des Zusammenbackens in die trocken gemischte Mischung einzuarbeiten.

9. Verfahren zum Herstellen einer Herbizidlösung, bestehend im wesentlichen aus den Schritten:

Zugeben einer trockenen pulverförmigen herbiziden Zusammensetzung nach Anspruch 1 zu einem Volumen Wasser;

Zugeben einer ausreichenden Menge der pulverförmigen trokken gemischten Mischung des herbiziden Mittels und des Phosphat-Solubilisierungsmediums zu dem Volumen Wasser, um zu bewirken, dass die in dem Wasser gelöste pulverförmige trocken gemischte Mischung ungefähr 0,1 Gew.-% bis 2-1/2 Gew.-% des herbiziden Wirkstoffs in der herbiziden Lösung bereitstellt; und

Bewegen des das herbizide Mittel und das Solubilisierungsmedium enthaltenden Wassers für eine ausreichende Zeitdauer, um eine substantielle Auflösung des Mittels und des Mediums in dem Volumen Wasser zu bewirken.

Revendications

1. Composition pulvérulente herbicide hydrosoluble sèche consistant essentiellement en :

un mélange mélangé à sec de
une première quantité de particules pulvérulentes sèches d'un agent herbicide sensiblement solide choisi dans le groupe consistant en l'acide 3,6-dichloropicolinique, l'acide 3,5,6-trichloro-2-pyridyloxyacétique, l'acide 4-amino-3,5,6-trichloropicolinique, l'acide 3,7-dichloro-8-quinoléinecarboxylique, le 2,2-dioxyde de 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one, le 3,5-dibromo-4-hydroxybenzonitrile, le 3,5-diiodo-4-hydroxybenzonitrile, chacun desquels n'étant pas très soluble dans l'eau, seul ou en combinaison entre eux, et
une seconde quantité de particules pulvérulentes solides sèches choisies dans le groupe consistant en le phosphate diammonique, le phosphate dipotassique, le phosphate disodique, le phosphate trisodique, le phosphate tripotassique et leurs mélanges comme milieu de solubilisation pour l'agent herbicide,
lesdites première et seconde quantités d'agent herbicide et de milieu de solubilisation étant les constituants principaux de la composition,
au moins environ 1 mol de milieu de solubilisation étant présente par mole d'agent herbicide,
ledit agent herbicide et le milieu de solubilisation ayant été mélangés à sec sous forme pulvérulente sans modifier l'état physique des particules pour conserver le caractère particulaire discret de chacune desdites première et seconde quantités desdites particules, et en l'absence de réaction chimique entre ledit agent herbicide et ledit milieu de solubilisation pour former un mélange sec relativement uniforme de ceux-ci,
une quantité suffisante de milieu de type phosphate dans le mélange mélangé à sec par rapport à la quantité d'agent herbicide combiné avec celui-ci étant présente pour que le mélange pulvérulent mélangé à sec se dissolve dans l'eau au cours de la préparation à partir de celui-ci d'une solution herbicide qui contient une proportion adéquate d'agent herbicide pour donner d'environ 0,1 à environ 2-1/2 % en masse d'agent herbicide actif dans la solution herbicide.

2. Herbicide selon la revendication 1 où ledit milieu de solubilisation sec est le phosphate diammonique.

3. Herbicide selon la revendication 1 où ledit milieu de solubilisation sec est le phosphate dipotassique.

4. Herbicide selon la revendication 1 où ledit milieu de solubilisation sec est le phosphate trisodique.

5. Herbicide selon la revendication 1 où ledit herbicide pulvérulent solide sec est le 3,5-diiodo-4-hydroxy-benzonitrile et le 3,5-dibromo-4-hydroxybenzonitrile.

6. Procédé de préparation d'une composition pulvérulente herbicide hydrosoluble sèche consistant essentiellement en les étapes de :

fourniture d'une première quantité de particules pulvérulentes sèches d'un agent herbicide sensiblement solide choisi dans le groupe consistant en l'acide 3,6-dichloropicolinique, l'acide 3,5,6-trichloro-2-pyridyloxyacétique, l'acide 4-amino-3,5,6-trichloropicolinique, l'acide 3,7-dichloro-8-quinoléinecarboxylique, le 2,2-dioxyde de 3-isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one, le 3,5-dibromo-4-hydroxybenzonitrile, le 3,5-diiodo-4-hydroxybenzonitrile, chacun desquels n'étant pas très soluble dans l'eau, seul ou en combinaison entre eux,
la fourniture d'une seconde quantité de particules pulvérulentes solides sèches de phosphate diammonique comme milieu de solubilisation pour l'agent herbicide,
lesdites première et seconde quantités d'agent herbicide et de milieu de solubilisation étant les constituants principaux de la composition,
au moins une mole de milieu de solubilisation étant présente par mole d'agent herbicide ; et

le mélange à sec de l'agent herbicide et du milieu de solubilisation sous forme pulvérulente sans modifier l'état physique des particules pour conserver le caractère particulaire discret de chacune desdites première et seconde quantités desdites particules, et en l'absence de réaction chimique entre ledit agent herbicide et ledit milieu de solubilisation pour produire un mélange sec relativement uniforme de ceux-ci,
une quantité suffisante du milieu de solubilisation de type phosphate étant présente dans le mélange mélangé à sec par rapport à la quantité d'agent herbicide combiné avec celui-ci pour amener le mélange mélangé à sec pulvérulent à se dissoudre sensiblement dans l'eau pendant la préparation par l'applicateur d'une solution herbicide qui contient une proportion adéquate d'agent herbicide pour donner d'environ 0,1 à environ 2-1/2 % en masse d'agent herbicide actif dans la solution herbicide.

7. Procédé selon la revendication 6 où est incluse l'étape de fourniture d'un mélange desdits agents actifs du point de vue herbicide pour le mélange à sec avec le milieu de type phosphate.

8. Procédé selon la revendication 6 où est incluse l'étape d'incorporation d'un agent antimottant dans le mélange mélangé à sec.

9. Procédé de préparation d'une solution herbicide consistant essentiellement en les étapes de :

addition à un volume d'eau d'une composition herbicide pulvérulente sèche selon la revendication 1 ;
addition d'une quantité suffisante du mélange mélangé à sec pulvérulent de l'agent herbicide et du milieu de solubilisation de type phosphate au volume d'eau pour amener le mélange mélangé à sec pulvérulent dissous dans l'eau à donner d'environ 0,1 % à environ 2-1/2 % en masse d'agent herbicide actif dans la solution herbicide ; et
agitation de l'eau contenant l'agent herbicide et le milieu de solubilisation pendant une durée suffisante pour réaliser une dissolution sensible de l'agent et du milieu dans le volume d'eau.